



Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing Thermal Barrier Coatings

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Prepared for the
27th Annual Cocoa Beach Conference and Exposition on
Advanced
Ceramics and Composites
sponsored by the American Ceramic Society
Cocoa Beach, Florida, January 26–31, 2003

National Aeronautics and
Space Administration

Glenn Research Center

Acknowledgments

This work was supported by the Ultra-Efficient Engine Technology (UEET) program, NASA Glenn Research Center, Cleveland, Ohio. Thanks to R. Pawlik for mechanical testing and G. Leissler for the preparation of thermal barrier coating material.

This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

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Presented at the 27th Annual Cocoa Beach Conference on
Advanced Ceramics and Composites
January 26-31, 2003
Cocoa Beach, Florida

[Paper Number: ECD-S2-14-2003 (Invited)]

Abstract

Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing Plasma-Sprayed Thermal Barrier Coatings

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Strength, fracture toughness and fatigue behavior of free-standing thick thermal barrier coatings of plasma-sprayed ZrO_2 -8wt% Y_2O_3 were determined at ambient and elevated temperatures in an attempt to establish a database for design. Strength, in conjunction with deformation (stress-strain behavior), was evaluated in tension (uniaxial and trans-thickness), compression, and uniaxial and biaxial flexure; fracture toughness was determined in various load conditions including mode I, mode II, and mixed modes I and II; fatigue or slow crack growth behavior was estimated in cyclic tension and dynamic flexure loading. Effect of sintering was quantified through approaches using strength, fracture toughness and modulus (constitutive relations) measurements. Standardization issues on test methodology also was presented with a special regard to material's unique constitutive relations.

Contents

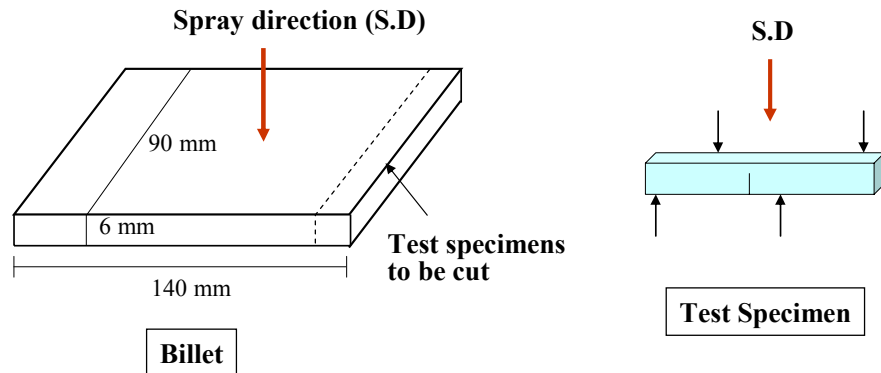
- I. Background**
 - II. Processing**
 - III. Strength**
 - IV. Fracture toughness**
 - V. Fatigue/slow crack growth**
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I. Backgrounds

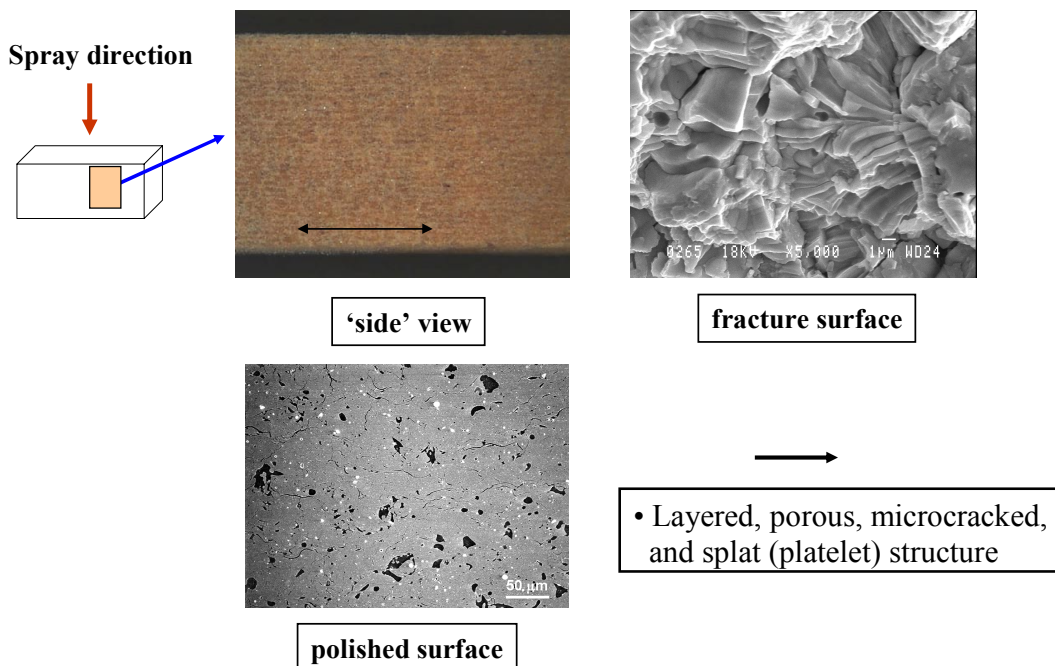
- **Thermal Barrier coatings (TBCs), ZrO_2 -8 wt% Y_2O_3 – important coating materials due to low thermal conductivity, high thermal expansivity, and unique microstructure**
 - **Somewhat anisotropic nature of porosity, microcracks and splat structure - a challenge in routine mechanical testing and data interpretation**
 - **Mechanical testing for TBCs performed to characterize strength, fracture toughness, fatigue, and deformation, and also to establish database**
 - **Results of mechanical testing presented and discussed, and related issues discussed**
-

II. Material Processing

- **ZrO₂-8 wt% Y₂O₃ powder** with an average particle size of 60 μm
- Plasma sprayed on a steel or graphite substrate
- SULZER-METCO ATC-1 plasma coating system with a 6-axes industrial robot used
- **Free standing** TBC billets fabricated
- Test specimens machined from billets with appropriate configurations
- Typical billets:

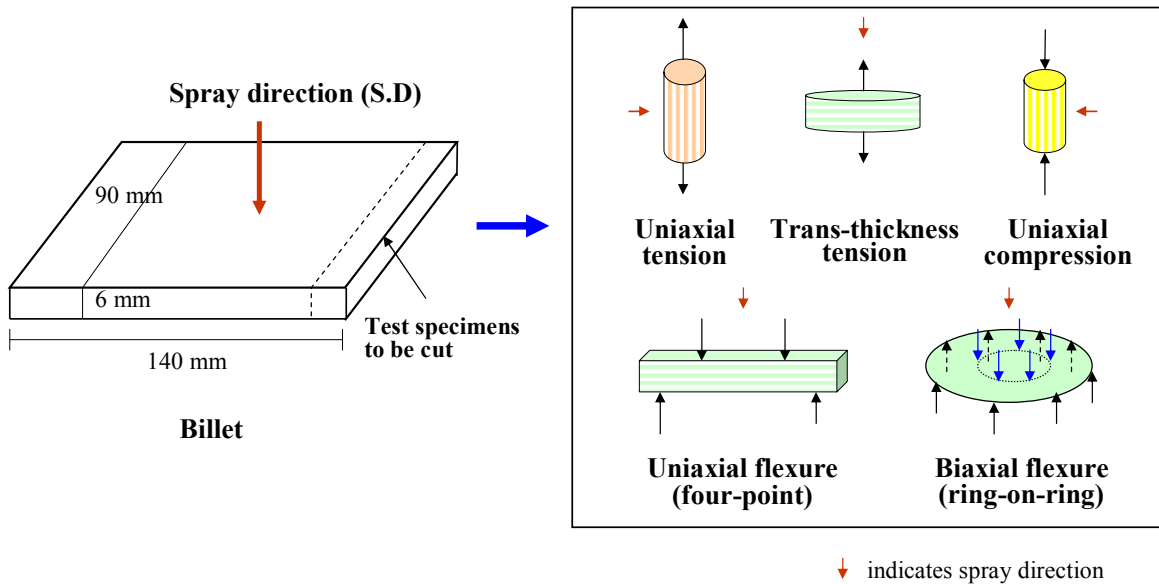


Unique Microstructure of TBCs



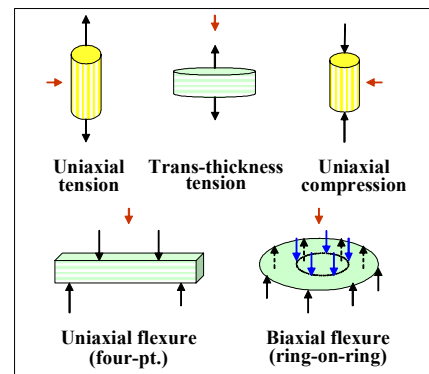
III. Strength Testing

Types of Testing/Test Specimens/Orientations



Test Matrix (strength)

Type of tests	Specimen geometry	No. of test specimens	Direction of fracture*
Uniaxial tension	15mm x 5mm ϕ	10	P
Trans-thickness Tension	15 mm x 3 mm (t) (diameter x thick.)	10	N
Uniaxial compression	10mm x 5mm ϕ	10	P
Uniaxial flexure (four-point)	3mm x 4mm x 25mm [10/20 mm spans]	30	P
Biaxial flexure (ring-on-ring)	25mm x 3mm (t) [11/22 mm rings]	10	P



* indicates the direction of fracture w.r.t plasma-spray direction.
Test temperature: ambient temperature in air.

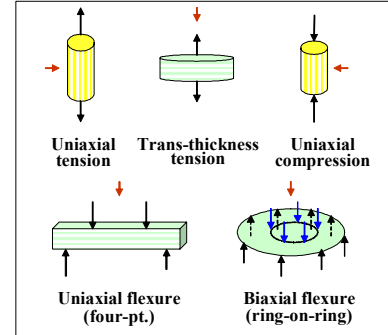
Experimental Results (strength)

Type of tests	No. of test specimens valid	Direction*	Average strength (MPa)#	Weibull modulus
Uniaxial tension	3	P	15(1)	-
Trans-thickness Tension	10	N	11(1)	13
Uniaxial compression	10	P	300(77)	4
Uniaxial flexure (four-point)	30	P	33(7)	6
Biaxial flexure (ring-on-ring)	10	P	40(4)	12

* indicates fracture direction w.r.t plasma-spray direction:

N: normal; P: parallel

represents ± 1.0 standard deviation



↓ indicates spray direction

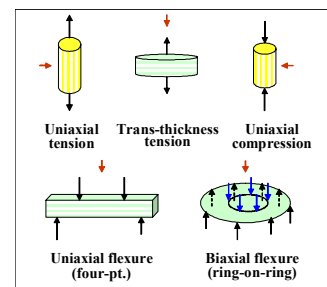
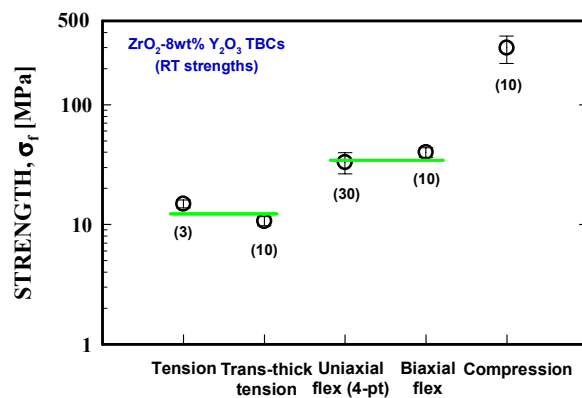
→ A basic assumption in strength calculation: a **continuum mechanics** (isotropic and linear-elastic)

$\sigma_f = 10\text{-}15$ MPa in tension
 $= 30\text{-}40$ MPa in flexure
 $= 300$ MPa in compression

Choi, Zhu, and Miller ('98,'99,'00,'01)

Experimental Results (strength)

Strength vs Type of Tests



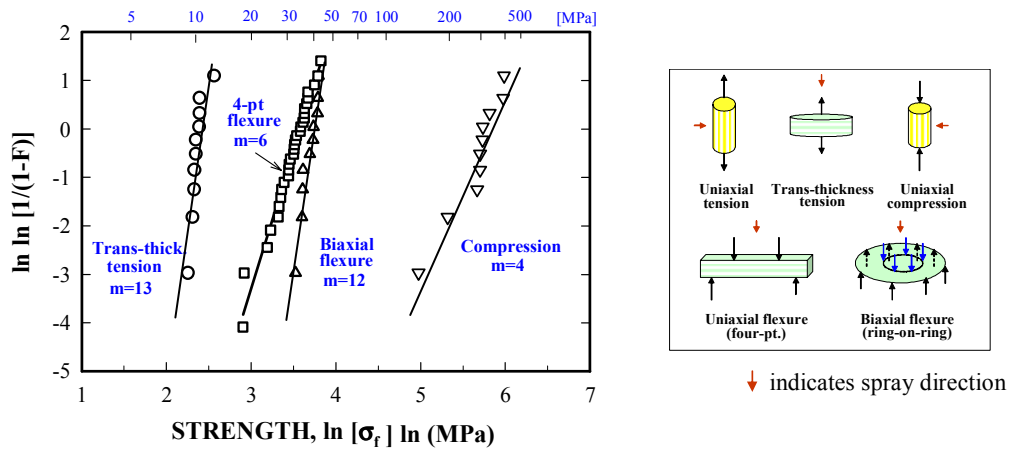
↓ indicates spray direction

→ $\sigma_f = 10\text{-}15$ MPa in tension
 $= 30\text{-}40$ MPa in flexure
 $= 300$ MPa in compression

The numbers indicates the number of specimens tested valid

Experimental Results (strength)

Weibull Strength Distributions

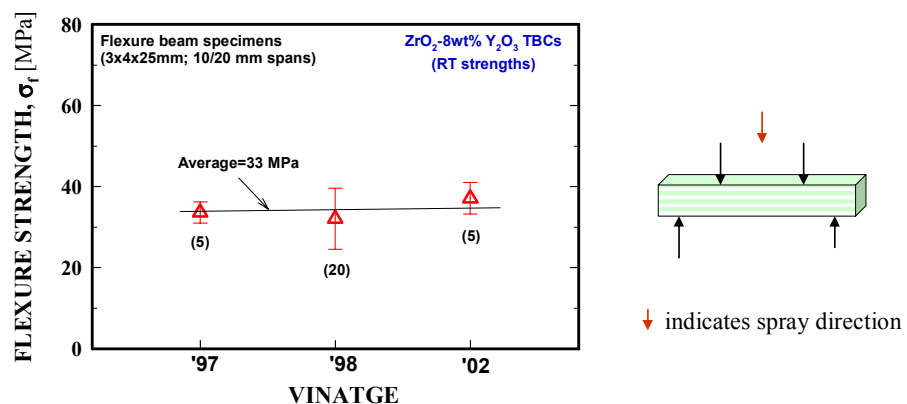


- Weibull moduli of $m=5-15$, a typical range for many commercial or in-house (dense) monolithic ceramics

Choi, Zhu, and Miller ('98,'99,'00,'01)

Experimental Results (strength)

Flexure Strength vs Vintage



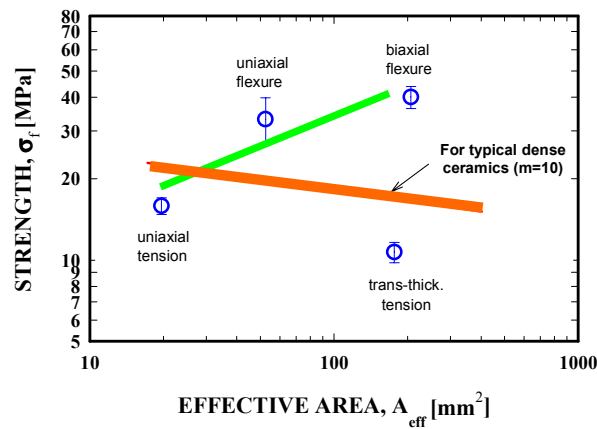
- Flexure strength – **less influence by vintage**, indicating consistency in plasma-spray processing over the years

Choi, Zhu, and Miller ('98,'99,'03)

The numbers indicates the number of specimens tested valid

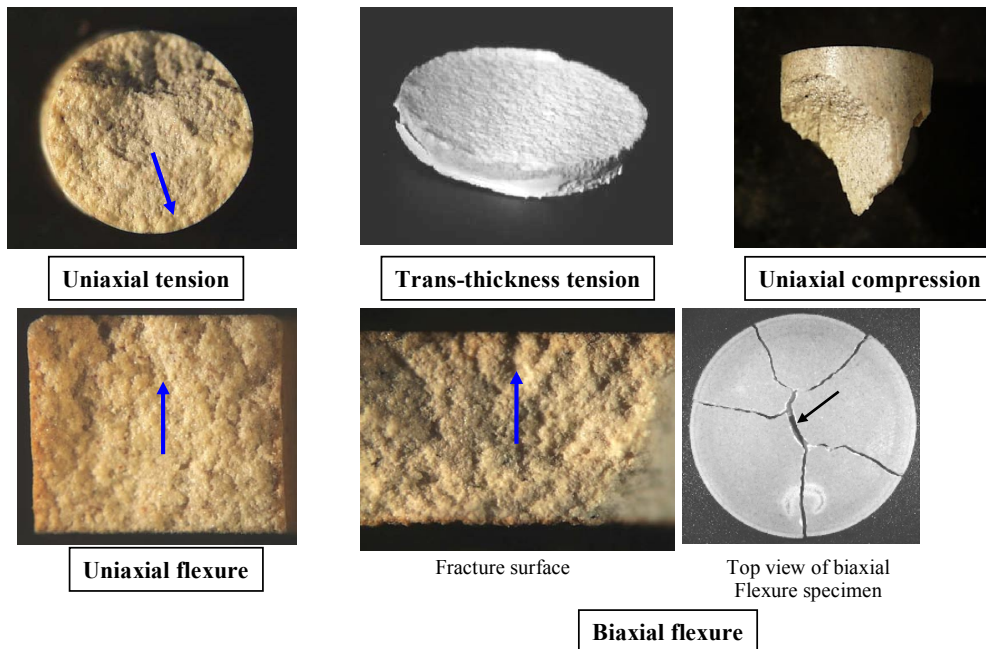
Strength vs. Effective Area – Size Effect

Strength-Effective Area (Weibull PIA model)



- **No reasonable agreement** in size effect between data and Weibull analysis (e.g., PIA); inconsistency in flaw populations (?)

Fractography (strength)

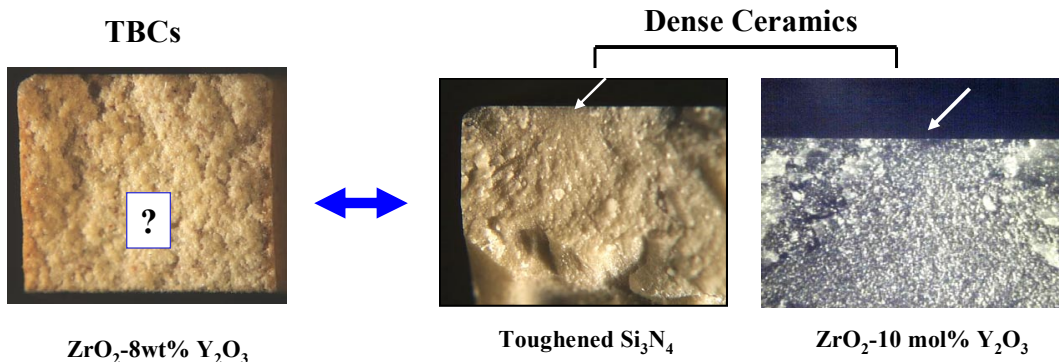


- **Very difficult** to locate fracture origins and to analyze their nature

Choi, Zhu, and Miller ('98,'99,'00,'01)

Fractography – A Great Challenge

Four-point flexure



Fracture mirror size (r_m):

- TBCs - 3-40 mm (estimated)
- Dense ceramics – 50-500 μm

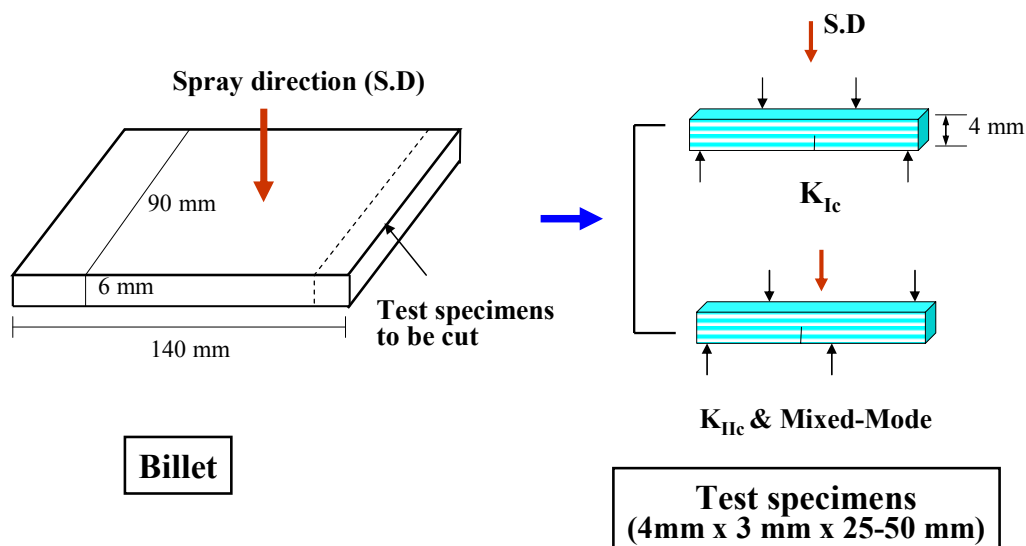
$$\sigma_f r_m^{1/2} = A$$

- Big mirror size & porous/microcracked nature of TBCs
→ An enormous **challenge** in fractography

Choi, Zhu, and Miller ('00,'01)
Choi ('02); Choi and Narottam ('02)

IV. Fracture Toughness Testing (Mode I, Mode II and Mixed Mode)

Types of Testing/Test Specimens/Orientations



Experimental (fracture toughness) (Mode I, Mode II and Mixed Mode)

Types & Procedures

• Sharp precracks generated

- Single edge v-notched beam (SEVNB) method:
Saw-notched → a sharp V-notch generated
with a razor blade with diamond paste, $a/W \approx 0.5$

• Test fixture configurations

- $A/B=10/5$ (typical); $s=0-3.6$ mm in mixed mode
- 10/20 or 20/40 mm spans in K_{Ic}

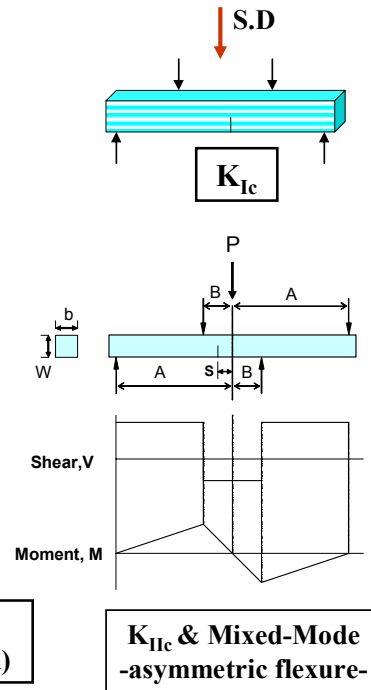
• Test temperatures

25 and 1316 °C in air

• Number of test specimens: typically ≥ 4

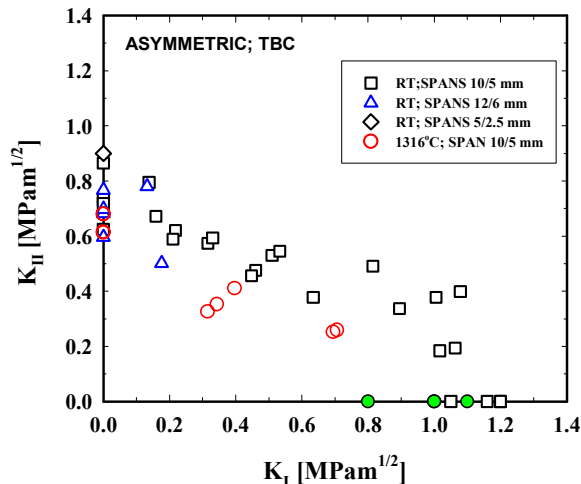


A typical, sharp SEVNB precrack (root radius $\leq 30 \mu\text{m}$)



Experimental Results (fracture toughness)

• Mode I, Mode II, and Mixed Mode (25 and 1316 °C)



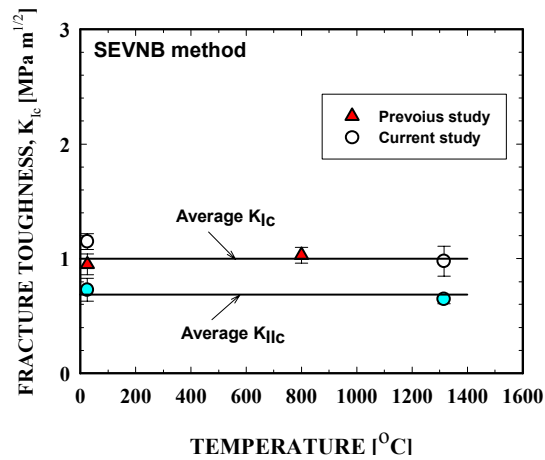
Test Temp(°C)	No. of specimens used	K_{Ic} (MPa $\sqrt{\text{m}}$)	K_{IIc} (MPa $\sqrt{\text{m}}$)
25	4 in K_{Ic} 9 in K_{IIc}	1.15(0.07)	0.73(0.10)
1316	4 each	0.98(0.13)	0.65(0.04)

- $K_{Ic} > K_{IIc} \rightarrow K_{IIc}/K_{Ic}=0.64$ & 0.66 (at 25 & 1316 °C)
- K_{Ic} and K_{IIc} at 25 °C $\geq K_{Ic}$ and K_{IIc} at 1316 °C
- **Elliptical relation** between K_I and K_{II}
- Test spans **independent**

Choi, Zhu, and Miller ('03)

Experimental Results (fracture toughness)

• Fracture Toughness vs. Temperature

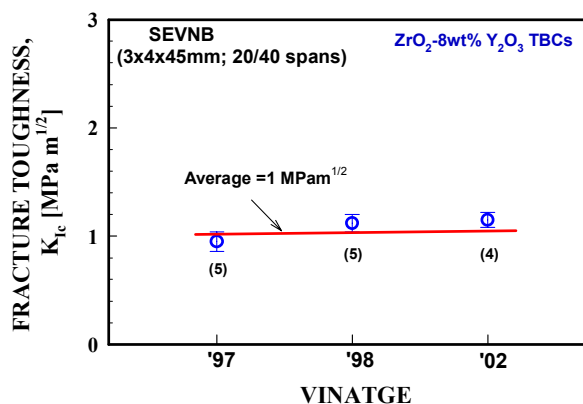


- Temperature **insensitive** in K_{Ic} and K_{IIc}
→ $K_{Ic} \approx 1$ and $K_{IIc} \approx 0.65 \text{ MPa}\sqrt{\text{m}}$
- $K_{IIc} / K_{Ic} \approx 0.65$

Choi, Zhu, and Miller ('98,'03)

Experimental Results (fracture toughness)

• Fracture Toughness (RT) vs. Vintage

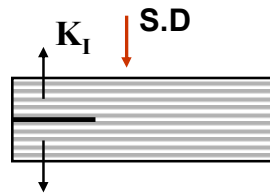


- Fracture toughness (K_{Ic}) – **less influence by vintage** (similar to strength), indicating consistency in plasma-spray processing over the years

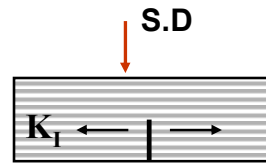
Choi, Zhu, and Miller ('98,'03)

Experimental Results (fracture toughness)

Fracture Toughness vs. Orientation



DCB specimens



SEVNB specimens

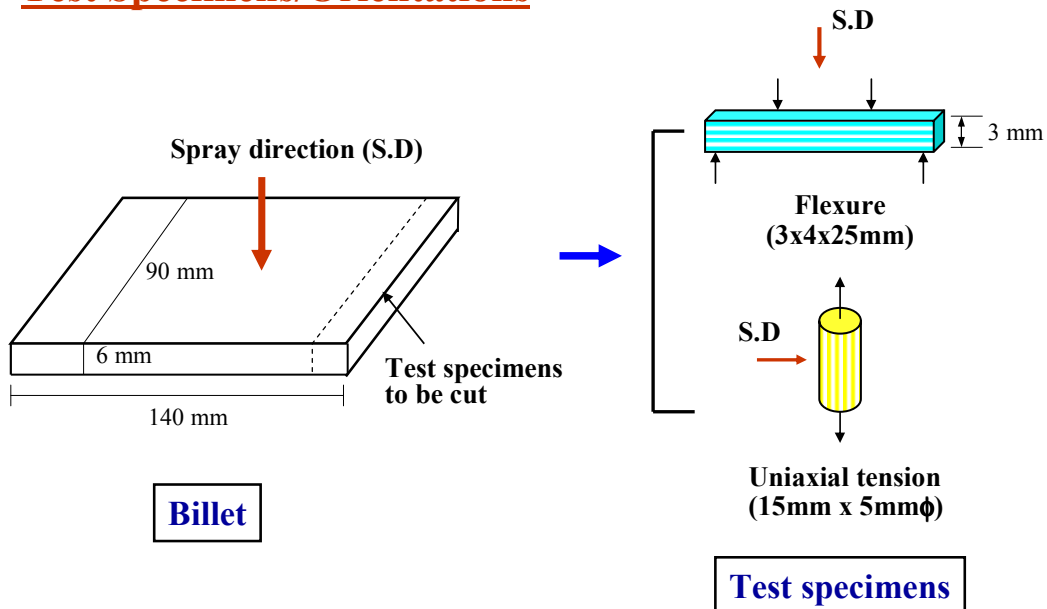
Direction of crack	Fracture Toughness K_{IC} (MPa \sqrt{m})	Method
Parallel to plasma spray direction	1.15±0.07	SEVNB (regular method)
Normal to plasma spray direction	1.04±0.05	DCB (Double Cantilever Beam)

- No significant difference in K_{IC} -- **Little directionality** effect on K_{IC}

Choi, Zhu, and Miller ('98,'03)

V. Fatigue/Slow Crack Growth

Test Specimens/Orientations



Experimental (fatigue)

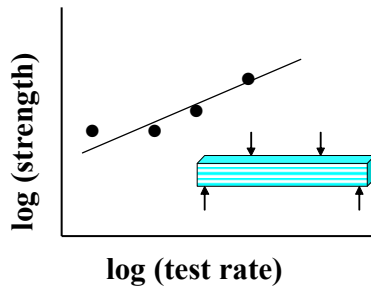
Test Types and Conditions

- Dynamic fatigue (ASTM C1425)

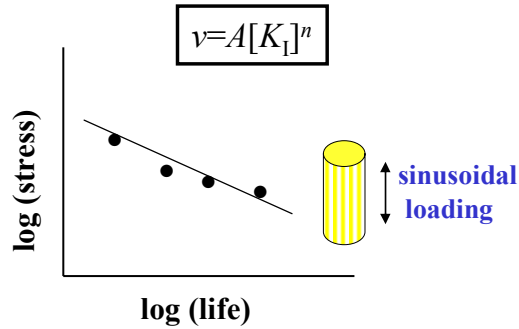
800 °C in air; 3 test rates in flexure

- Tensile cyclic fatigue

RT in air; sinusoidal; $R = 0.1$; $f = 10$ Hz



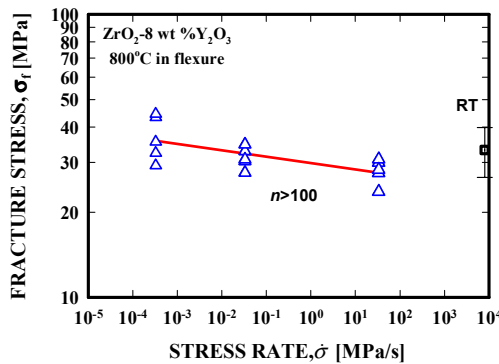
Dynamic fatigue



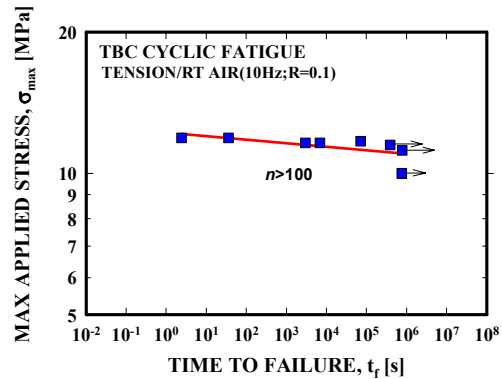
Tensile cyclic fatigue

Experimental Results (fatigue/SCG)

Dynamic fatigue



Tensile cyclic fatigue



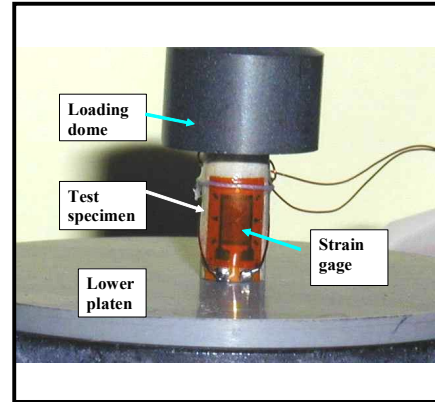
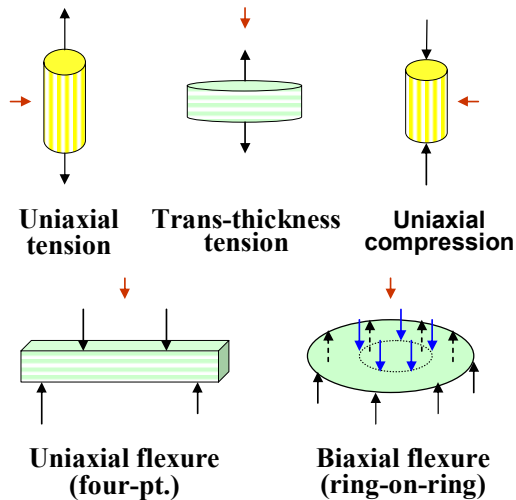
$$v = A[K_I]^n$$

- Slow crack growth (SCG) parameter n :
 $n > 100$ in both dynamic and tensile cyclic fatigue –
Little susceptibility to SCG (fatigue)

Choi, Zhu, and Miller ('98,'99,'01)

VI. Deformation (Stress-Strain) Behavior

5 Specimen/Loading Conditions Considered

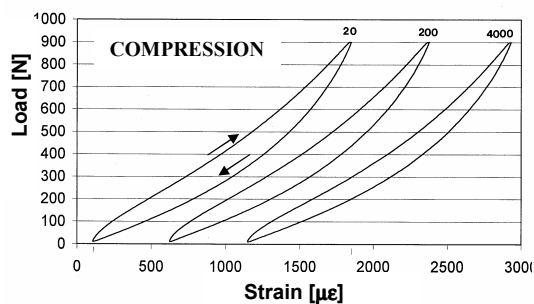
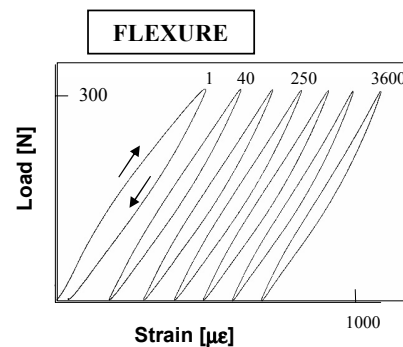
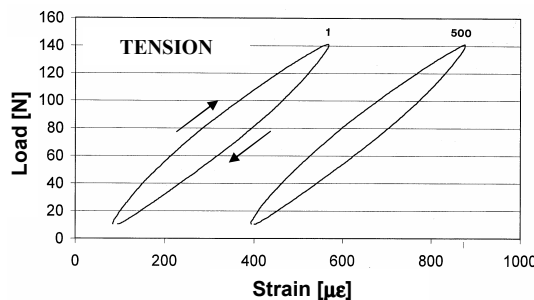


Test Setup (strain gaging)

↓ indicates spray direction

Experimental Results (deformation)

Typical Load-Strain Curves

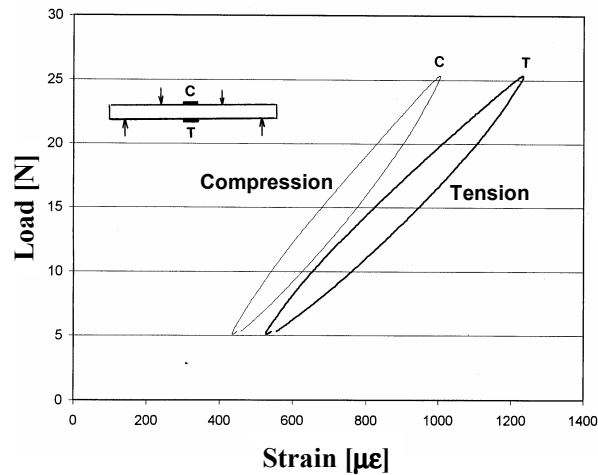


- **Non-linearity** with **hysteresis** but **elastic** -desirable in TBCs but difficulty in analysis
- Independent of the number of cycles and test rate (not-viscoelastic)

Choi, Zhu, and Miller ('00,'01)

Experimental Results (deformation)

Four-Point Flexure

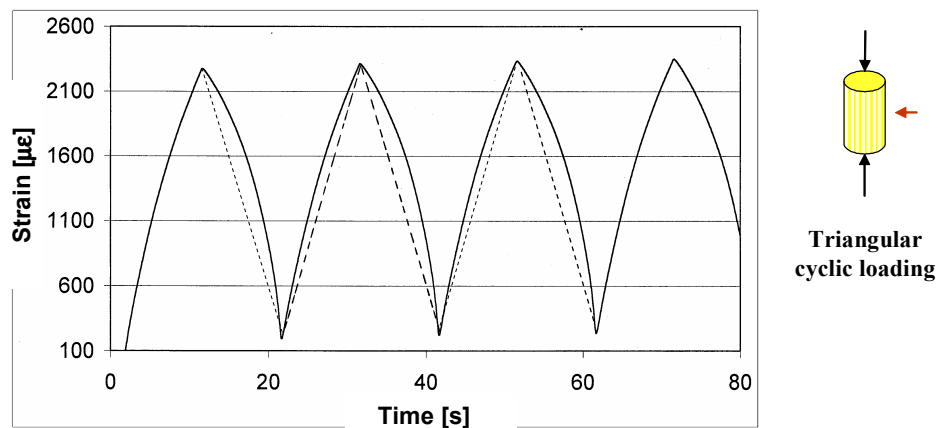


- **Different response of strain** in compression and tension
- A possible **neutral axis shift** due to different elastic modulus
- Flexure stress calculation – complex

Choi, Zhu, and Miller ('01)

Experimental Results (deformation)

Response of Output Wave Form to Cyclic Compression Loading

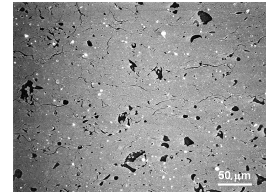
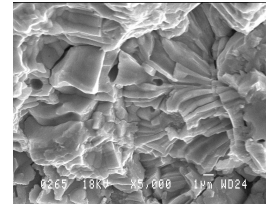
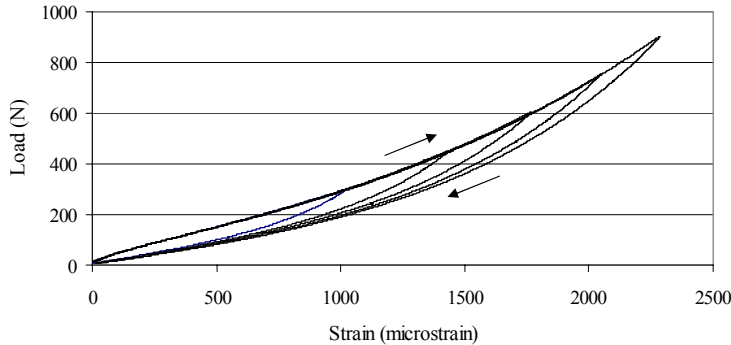


→ The output wave form - **distorted** from the input triangular wave form

Choi, Zhu, and Miller ('01)

Deformation (Stress-Strain) Behavior

What is the cause of nonlinearity and hysteresis?



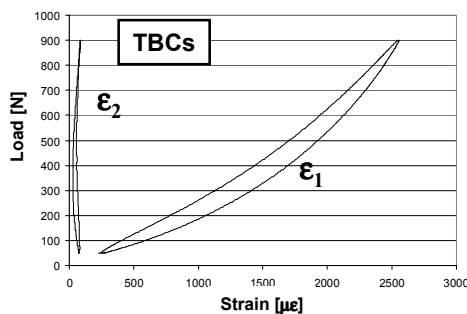
Major reason – **‘loosely’ connected open structure** due to pores and microcracks

- Internal friction and densification
- Still overall elastic behavior

Eldridge, Morscher, and Choi ('02)

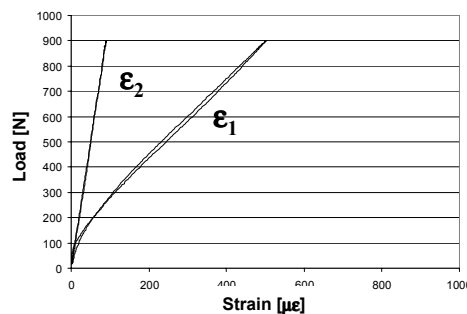
Deformation (Stress-Strain) Behavior

‘Loosely-Connected Open’ Structure – Poisson’s Response



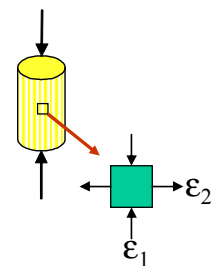
Exhibits **no or little change** in lateral strain: Poisson’s ratio not well defined

Open, loose structure



Exhibits a **linearly increasing** lateral strain: Poisson’s ratio well defined

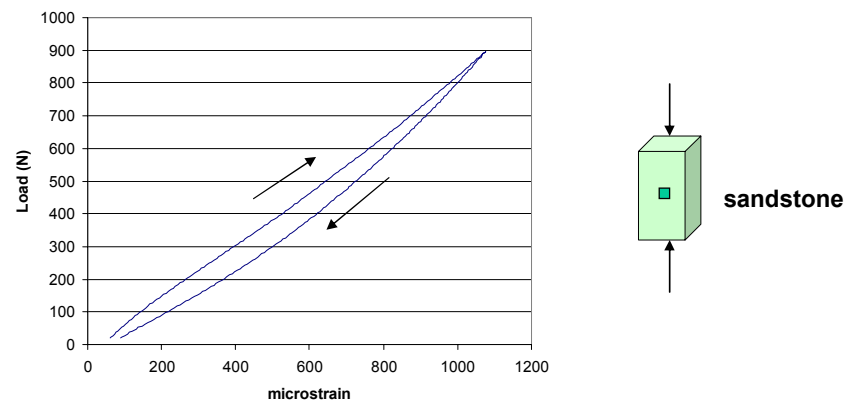
Dense structure



Poisson’s ratio
 $\nu = |\epsilon_2 / \epsilon_1|$

Experimental Results (deformation)

Sandstone – Another Example of Open Structure

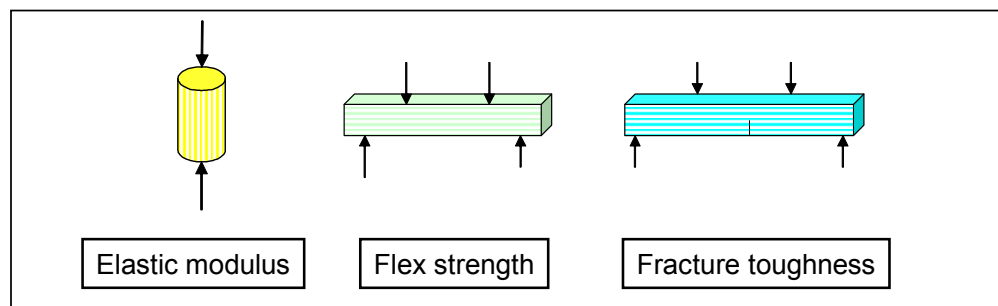


→ Open, loose structure: non-linearity with hysteresis
-- Similarity to TBCs

VII. Sintering – A Changer of Structure

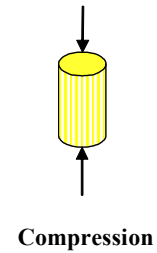
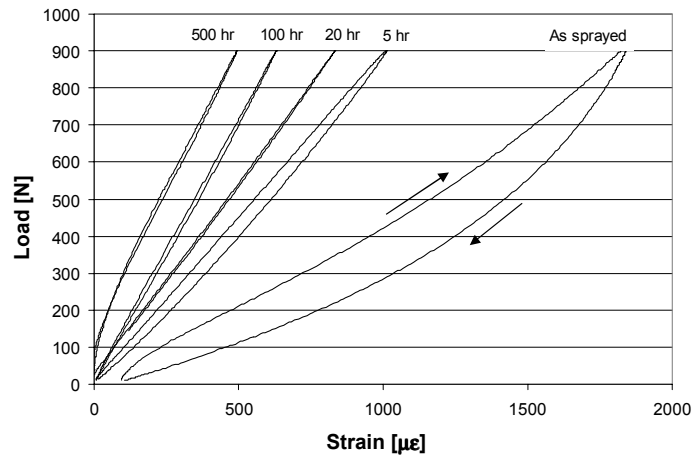
Sintering conditions:

- Temperature/environment: 1316 °C/air
- Annealing time: 0, 5, 20, 100, and 500 h
- Determine as a function of anneal time:
 - Elastic modulus
 - Fracture toughness (K_{Ic})
 - Flexure strength
 - Thermal conductivity



Experimental Results (sintering)

Elastic Modulus

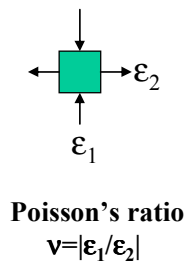
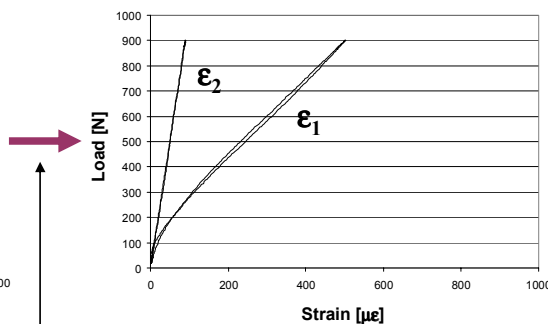
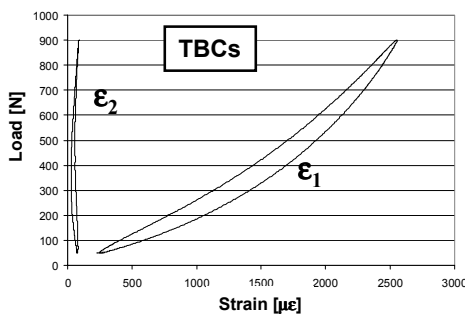


- Slope (elastic modulus) **increases** with anneal time
- Linearity **increases** with anneal time
- Hysteresis **decreases** with anneal time

→ Implies a change of microstructure from 'loosely' connected to 'closely' connected

Experimental Results (sintering)

Well-Developed Poisson's (Lateral Strain) Response

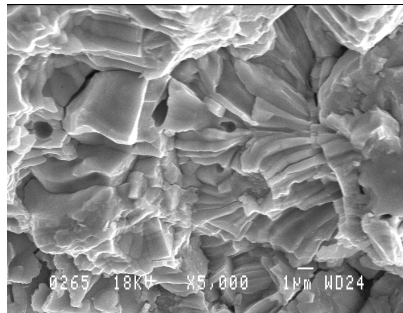


500 h annealing

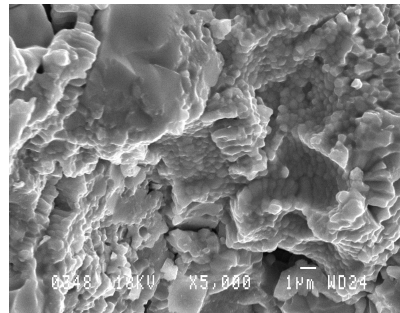
Open structure → More closely-connected structure

Experimental Results (sintering)

Microstructure



As-sprayed

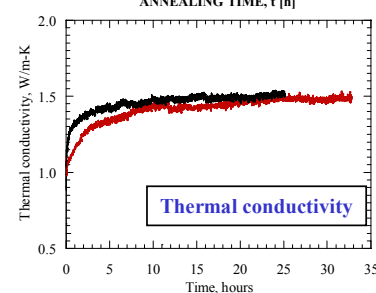
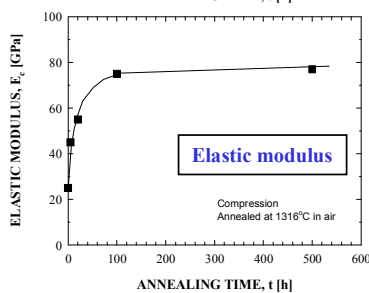
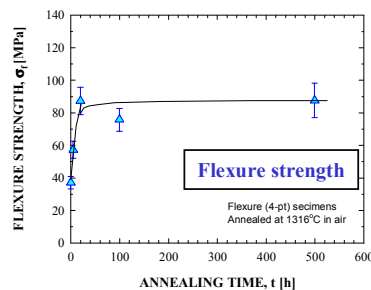
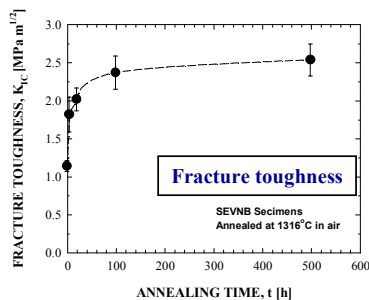


100 h annealed

- **As-sprayed** - Large amounts of **microcracks and pores** with a unique platelet (splat) structure presented
- **100 h annealing** - **Increased grain growth** at longer annealing time

Experimental Results (sintering)

- **Summary on elastic modulus, flexure strength, fracture toughness and thermal conductivity**



→ Properties change **exponentially** with sintering time

Choi, Zhu, and Miller ('03)

Standardization Issues

- **The most hindering factor** in establishing test methods for as-sprayed TBCs: **non-linearity** & **hysteresis** in the constitutive relations
 - Flexure testing (uniaxial and biaxial) maybe inappropriate due to difference in modulus between tension and compression
 - Poisson's ratio not well-defined
 - Impulse excitation technique maybe inappropriate
 - Pure tension and compression testing – impose less problems
 - Fracture toughness testing – maybe OK in view of low fracture loads
 - Fractography – challenging
 - Properties change with sintering/service conditions
 - requires to evaluate based on sinter/service conditions
-

Summary

- **Strength:**
tension: 10-15 MPa; flexure: 30-40 MPa; compression: 300 MPa
Weibull modulus: 5-15
 - **Fatigue/Slow Crack Growth:**
SCG parameter $n > 100$
 - **Fracture Toughness:**
 $K_{Ic} = 1.0 \text{ MPa}\sqrt{\text{m}}$ up to 1316 °C
 $K_{IIc} = 0.7 \text{ MPa}\sqrt{\text{m}}$ up to 1316 °C
 - **Deformation:**
nonlinear elasticity with hysteresis;
imposes problems in continuum approach (*test standards*)
 - **Sintering:**
significant influence - a changer of most properties!
-

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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE July 2003		3. REPORT TYPE AND DATES COVERED Technical Memorandum
4. TITLE AND SUBTITLE Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing Thermal Barrier Coatings			5. FUNDING NUMBERS WBS-22-714-04-05	
6. AUTHOR(S) Sung R. Choi, Dongming Zhu, and Robert A. Miller				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-14076	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2003-212516	
11. SUPPLEMENTARY NOTES Prepared for the 27th Annual Cocoa Beach Conference and Exposition on Advanced Ceramics and Composites sponsored by the American Ceramic Society, Cocoa Beach, Florida, January 26-31, 2003. Sung R. Choi, Ohio Aerospace Institute, Brook Park, Ohio 44142; Dongming Zhu, U.S. Army Research Laboratory, NASA Glenn Research Center; and Robert A. Miller, NASA Glenn Research Center. Responsible person, Sung R. Choi, organization code 5920, 216-433-8366.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 07 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Strength, fracture toughness and fatigue behavior of free-standing thick thermal barrier coatings of plasma-sprayed ZrO_2 -8wt % Y_2O_3 were determined at ambient and elevated temperatures in an attempt to establish a database for design. Strength, in conjunction with deformation (stress-strain behavior), was evaluated in tension (uniaxial and trans-thickness), compression, and uniaxial and biaxial flexure; fracture toughness was determined in various load conditions including mode I, mode II, and mixed modes I and II; fatigue or slow crack growth behavior was estimated in cyclic tension and dynamic flexure loading. Effect of sintering was quantified through approaches using strength, fracture toughness, and modulus (constitutive relations) measurements. Standardization issues on test methodology also was presented with a special regard to material's unique constitutive relations.				
14. SUBJECT TERMS Thermal barrier coatings; Strength; Fracture toughness; Fatigue; Deformation; Sintering effect			15. NUMBER OF PAGES 26	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

